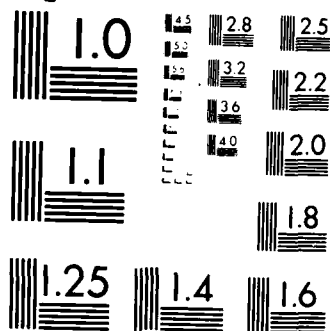


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# CEMENTITIOUS MIXTURES FOR SEALING EVAPORITE AND CLASTIC ROCKS IN A RADIOACTIVE-WASTE REPOSITORY

by

Lillian D. Wakeley

Structures Laboratory

DEPARTMENT OF THE ARMY

Waterways Experiment Station, Corps of Engineers  
PO Box 631, Vicksburg, Mississippi 39180-0631

and

Della M. Roy

Materials Research Laboratory  
The Pennsylvania State University  
University Park, Pennsylvania 16802



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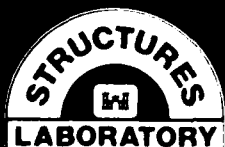
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Office of Nuclear Waste Isolation  
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  Cement-based mixtures were proportioned for sealing a potential geologic repository for high-level radioactive waste in evaporite and related rock strata. The slightly expansive cementitious mixtures were based on Class H portland cement and Class C fly ash, with silica flour and calcium sulfate additives, and a low ratio of water to cementitious solids (w/s = 0.32). Both a salt-free and a salt-containing version were used in tests with anhydrite, siltstone, and halite.		

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20. ABSTRACT (Continued).

Although it is difficult to achieve strong bonding between anhydrite and cement-based mixtures, tensile bond strengths, interface permeability, and other properties of the composites as a whole indicate achievement of the desired chemical and physical compatibility between these materials. Integrity of bonding and low permeability to water (on the order of  $10^{-6}$  darcy) suggest the likelihood of attaining durable sealing of repository access shafts and boreholes through the use of such chemically tailored cementitious mixtures.

Rock samples used in these sealing studies were obtained from various areas throughout the United States. The data in the present paper represent results for rocks obtained from the Palo Duro Basin, TX, and, as such, typify bedded-salt strata.

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## Preface

This work was supported at the Materials Research Laboratory of the Pennsylvania State University (PSU) by the US Department of Energy (DOE), contract No. DE-AC02-83CH10140, Subcontract E512-04200 with Battelle Project Management Division, Office of Nuclear Waste Isolation (ONWI). J. B. Moody and D. P. Moak were ONWI project managers.

Della M. Roy was principal investigator at PSU for the research reported herein, with participation by L. D. Wakeley, M. W. Grutzeck, P. H. Licastro, and A. Das.

The report was prepared at PSU and in the Concrete Technology Division (CTD) of the Structures Laboratory (SL) of the US Army Engineer Waterways Experiment Station (WES) under the direction of Mr. J. M. Scanlon, Chief, CTD, and Mr. B. Mather, Chief, SL, by L. D. Wakeley. Publication costs were provided by Sandia National Laboratories.

Commander and Director of WES during the conduct of this investigation and preparation of this report was COL Robert C. Lee, CE; Technical Director was Mr. Fred R. Brown. During the publication of this report, COL Allen F. Grum, USA, was Director of WES; Dr. Robert W. Whalin was Technical Director.

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# CEMENTITIOUS MIXTURES FOR SEALING EVAPORITE AND CLASTIC ROCKS IN A RADIOACTIVE-WASTE REPOSITORY

## Introduction

1. The Palo Duro Basin of northern Texas is one of seven potentially favorable locations for a mined geologic repository for disposal of commercial or defense high-level radioactive wastes and spent fuel (Office of Nuclear Waste Isolation (ONWI) 1983a). Shafts and boreholes, as disturbances through the host rock and overlying strata, are a potential weakness in guaranteeing waste isolation. Cementitious mixtures were proportioned for use in sealing such an underground repository in evaporite strata (ONWI 1983b). For this study, evaporite rocks from the Palo Duro Basin, which are typical of bedded-salt deposits throughout the United States, were used. The dryness, plasticity, thickness, and lateral continuity of halite rock are desirable properties for repository-host strata (Stone and Webster Engineering Corp. 1983).

2. An adequate repository-sealing system will permanently seal man-made disturbances. The system will include cementitious materials, rock used as backfill or aggregate, the host rocks and associated groundwater, and other appropriate materials. The variety of rock types encountered down the length of a shaft will define a broad range of conditions, due to differences in strength, porosity, permeability, salt or water content, total chemical composition, and other critical properties. Sealing will require cementitious materials selected for compatibility with each rock type, including carbonate and poorly indurated clastic rocks often overlying and within the evaporite rocks.

3. The interfacial region between cement and host rock is currently considered the most important element of the seal, presenting special challenges to characterization. Water solubility of the evaporites and poor induration (softness) of associated clastic rocks limit the effectiveness of routine testing procedures used for either cement or rock.

## Mixture Proportioning

4. Cementitious mixtures used in these experiments were sanded grouts derived from the Bell Canyon Test (BCT)-family of materials (Roy, Grutzeck,

and Wakeley 1983, 1984), based on API Class H cement to control rate of heat release. Experiments with interfacial composites with anhydrite--the major nonhalite evaporite in the Palo Duro Basin--used a salt-free mixture. This mixture, MRL 83-06, contained similar proportions of sand and portland cement, a fine silica-flour additive to reduce free lime, and a low ratio of water to solids ( $w/s = 0.32$ ). Its sulfate content was higher than that of its BCT-antecedent, giving it a longitudinal expansion of about +0.06 percent at 10 days of curing at 38 C. Because of its workability, and density and impermeability when set, this mixture also was used in experiments with siltstone from the Palo Duro Basin (Alibates Formation). Components of this mixture are listed in Table 1.

5. An equivalent salt-containing mixture, designated 83-05 (Table 1), was developed for use in halite strata. This mixture was intended to expand during curing and aging by in situ alteration and hydration, to enhance bonding to the host rock.

6. Samples of the cementitious mixtures alone were mixed and cast according to ASTM procedures, cured at 38 C and >95 percent relative humidity, and tested by standard methods to establish baseline data for the composites. Data for physical and mechanical properties (Table 2) include some from samples of 83-06 cured in simulated groundwater (from nonevaporite strata in the Palo Duro Basin; MRL No. E33).

#### Properties of Rock Materials

7. In addition to the beds of relatively pure halite and anhydrite in the lower San Andres Formation, strata include beds and stringers of mixed clastic, carbonate, and evaporite lithologies. Many of the clastic units are naturally cemented with calcite, dolomite, or anhydrite, are porous and saturated, or are poorly indurated. Units with very low strength (<5.0 MPa in compression) could not be prepared for individual or composite tests of permeability or tensile strength.

8. The anhydrite, siltstone, and halite chosen for composites were derived from core segments from the No. 1 J. Friemel borehole, Deaf Smith County, TX. Anhydrite (MRL No. C76) and siltstone (MRL No. C79, 2360-m depth) were cut to a diameter of 25.4 mm. The coarse crystal size of the halite segments consistently caused fracture, in an unconfined state and under the pressure of

lab-scale drilling, preventing recoring. Porosity and permeability were measured for anhydrite and siltstone, as reported in Table 3.

9. Using small broken pieces of the halite or other rock to be studied, interface samples were cast in plastic vials 15 mm in diameter, with portions of larger batches of mortar mixed for other tests, to minimize batch-size variability. Bits of the rock were encased in mortar within these vials, and cured at 38 C and >95 percent relative humidity. The vial then was cut away from the cylinder, and very thin wafers of cement plus rock were cut on a small oil-cooled saw. Composite samples were prepared of mixture 83-05 with halite, anhydrite, and mudstone from the No. 1 J. Friemel rocks, and of 83-06 with anhydrite and siltstone. These samples were studied via scanning electron microscopy (SEM) and optical petrography.

#### Interfaces with mixture 83-05

10. Samples cast of the salt-containing mixture 83-05 with mudstone (C74), halite (C75), and anhydrite (C76) required more than 7 days to harden, and were studied qualitatively after at least 14 days. Samples cast with mudstone usually broke either through the rock or along the interface. Some samples separated at the interface during preparation of thin sections, leaving no bond to observe by this method. In a previous study (Wakeley and Roy 1983), similar problems had been encountered with comparable mudstone from the Dewey Lake Formation. The relatively low strength of the mudstone (around 20 MPa) and the presence of swelling clay minerals made it difficult to prepare small-scale samples to simulate restrained conditions of field performance.

11. Samples of mixture 83-05 and halite from the No. 1 J. Friemel core (C75) were successfully prepared for both SEM and optical petrography after curing from 28 to 90 days. In all cases--even grinding of thin sections--the bond between the cementitious material and the halite remained intact. SEM images (50 to 2000X) showed continuous bonding between halite and paste, with no visible cracks either along the interface or within either component (Figure 1).

12. The paste interfingered with the surface roughness of the rock, forming mechanical bonding, and surfaces of the halite did not appear to be washed out along crystal boundaries. As intended, the formulation of mixture 83-05 included sufficient NaCl to prevent significant dissolution of the halite host rock that could potentially destroy seal integrity.

13. Secondary-electron images from SEM and thin sections show that porosity of the grout may be higher within the first few hundred micrometres from the interface, or that the hydration products are less dense in this region. This observation agrees with similar research on other materials (Struble, Skalny, and Mindess 1980; Barnes, Diamond, and Dolch 1978). Images of chloride distribution show concentrations of chloride ions around the pores (Figure 2), but the source of these mobile ions may be the NaCl component of the grout, rather than the rock. A layer of crystal growth was locally visible along the interface, apparently cement hydration products (calcium silicate hydrate (C-S-H) and  $\text{Ca(OH)}_2$  were detected). Small fibrous crystals--possibly ettringite--had grown along cleavage planes in the halite, near and extending to the interface, and small fractures through the paste were filled with halite.

14. Although the mixture was intended for use with halite, it was important to determine whether the enhanced solubility of anhydrite in contact with brine would preclude bonding between the mixture and San Andres anhydrite. Thin sections of such composites showed partial separation along the interface, with attendant crystallization of halite in the voids created by this separation. The bond formed between 83-05 and anhydrite did not show growth of cement hydration products as a bonding agent. Interfacial voids could have resulted from minor but deleterious dissolution of anhydrite. SEM samples of this same combination--83-05 plus anhydrite--showed a surprisingly sharp contact between rock and grout. SEM images depicting distribution of sulfates and chloride in the samples show no apparent migration of sulfate from rock into grout, nor of chloride from grout into rock. The extremely low porosity of this dense anhydrite probably inhibited ion transfer across the interface.

#### Interfaces with mixture 83-06

15. Mixture 83-06 was a salt-free formulation, designed for plugging and sealing nonhalite strata, especially anhydrite. Composite samples, therefore, centered around anhydrite and siltstone.

16. The major difficulty encountered in preparing thin sections was that unrestrained samples left for only a few days at ambient lab conditions separated along the interface. The grout itself often appeared extensively cracked, in patterns that were not always related to the interfacial region. Both sulfur maps and secondary-electron images suggested that in this case, there was some sulfate migration, and crystal growth along the interface. This is in contrast with samples of 83-05 + anhydrite, in which neither of these appeared to have occurred.

17. Probably, the vacuum desiccation required within the SEM sample chamber damaged the interfacial bonding of these 83-06 samples. Samples prepared as thin sections fared somewhat better, and petrographic study revealed abrupt, unfractured interfacial bonds. The paste also displayed crystal growth of hydrated cement in a relatively uniform network, distinct from the darker unhydrated cement components. The difference between SEM and optical petrographic samples probably stems from the contrast in desiccation of the sample, which was far greater in SEM (magnification ranges overlapped).

18. Both thin sections and SEM samples of mixture 83-06 with siltstone (C79) showed mechanical bonding between rock and mortar. In some thin sections, the rock was cracked parallel to the interface, but a layer of the rock remained affixed to the cement. Related to the high permeability of this rock ( $>10^{-4}$  darcy) (one could give an SI equivalent using the formula darcies  $\times 9.869233 \text{ E} - 13 = \text{m}^2$ ) is a fairly rough surface texture, which allowed interpenetration of cement and rock on a micro scale. This high permeability also would have permitted relatively unencumbered ion migration across the interface.

#### Bond Strength and Permeability Between Mortars and Rocks

##### Preparation and testing of samples

19. Tensile strength of the bond between grout and rock materials is one indicator of chemical and physical compatibility between these materials. Rock samples were prepared as cylinders 25.4 mm in diameter and length, to fill half of a removable cylindrical mold. Roughness of the surface for bonding to the mortar was standardized by abrading.

20. The grout was cast onto rock cylinders in the molds, and cured for 14 to 90 days at 38 C and >95 percent relative humidity. Composites with anhydrite (C76) and siltstone (C79) were successfully prepared and tested for bond strength to mixture 83-06. In addition, bond strength was tested at 28 and 56 days for samples cured in a water formulated to represent nonsaline groundwater from the Palo Duro Basin (MRL No. E33) (Clark and Bradley 1983).

21. Bond strength for mixture 83-06 with anhydrite did not exceed 1.5 MPa (three to six specimens per test age). There are no strong trends in the data relative to curing time over the 90-day test period. For samples cured at high humidity, bond strength decreased with longer curing time. The

magnitude of this decrease is minimal, but the bond strengths at all curing times were lower than the tensile strength of the grout alone.

22. Bond strength of samples cured in E33 simulated groundwater approximately doubled between 28 and 56 days (from  $0.6 \pm 0.2$  to  $1.1 \pm 0.1$  MPa). This established a trend opposite to that of the samples cured at high humidity. However, the bond strengths for these samples were not notably higher than those of their counterparts cured at high humidity, even though the tensile strength of the grout alone, when cured in E33, was higher than its high-humidity counterpart (Table 4).

23. In all cases of composites with anhydrite, there was total separation of rock and mortar as a result of the tensile test, suggesting minimal bonding. A white crystalline powder, consisting of calcium hydroxide ( $\text{Ca(OH)}_2$ ) and gypsum, was present on both separated surfaces of all samples. The presence of this interfacial layer is consistent with observations made via SEM and optical microscopy.  $\text{Ca(OH)}_2$  apparently is the major hydrated phase in the interfacial region from the cement, usually formed at an early age. It imparts lower strength to cured pastes than the C-S-H component. At the interface, it may support some chemical bonding, but is mechanically weak.

24. Due to the paucity of well-indurated siltstone in the segments of No. 1 J. Friemel core available, interface samples were tested for bond strength at only two curing times. Values for bond strength are higher at 28 days than at 14 days of curing. Separation of rock from grout was not complete at testing: some of each component remained mechanically bonded to the opposite surface.

#### Permeability of Composite Samples

25. Interface samples of 83-06 and anhydrite were prepared for testing permeability to water, as described by Wakeley and Roy (1982). A major modification to that procedure was that samples were cast in removable brass molds, instead of permanent brass cylinders. Apparatus of the Hassler type used for testing permeability in this case confined the sample in a rubber sleeve under pressure that was greater than the driving pressure of the test fluid.

26. Samples were tested after 28 to 120 days of curing. In general, there were no obvious trends with curing time, but permeability remained at  $10^{-6}$  darcies or lower. After testing, a white deposit on the grout surface

traced the flow path through the sample, extending across the sample at the upper surface and tapering down the interface (Figure 3). In this case, the interface itself, known from X-ray diffraction (XRD) and petrography to include a layer of  $\text{Ca(OH)}_2$  plus gypsum, is a preferred pathway for water flow. Permeability of  $10^{-6}$  darcies may be within an acceptable range. Samples cured in E33 groundwater had no measurable permeability ( $<10^{-8}$  darcies) at any test age up to 56 days.

27. For the composites cured at high humidity, permeability of the interfacial region was greater than that of either the grout alone ( $<10^{-8}$ ) or the anhydrite rock ( $<10^{-8}$ ). In some cases, flow was consistent at about  $10^{-6}$  darcies for 1 day or more, and then increased sharply for the remainder of the test. Microscopic examination of these samples after testing showed that failure occurred by dissolution of anhydrite along the interface. The permeability of siltstone composites approximated that of the siltstone alone ( $>10^{-4}$  darcies).

#### Summary and Conclusions

28. Cement-based materials were developed to be part of a sealing system in a proposed repository for high-level radioactive wastes. Compatibility between cement and rock was defined through characterization of the rocks, the cementitious mixtures, and composite samples that include interfaces between the two components.

29. A slightly expansive mixture (83-06) intended for use with nonsalt evaporites and clastic rocks was cast in contact with anhydrite and siltstone. An equivalent salt-containing mixture was developed for use in halite strata in the same region, and was tested with halite rock taken from the same borehole.

30. Results of tests for tensile bond strength and permeability were considered to indicate chemical and physical compatibility. For the former, grout was cast against anhydrite and siltstone cylinders 25 mm in diameter and length. Strengths were low (less than 2 MPa), but compared favorably with strengths of similar samples prepared in other experiments (Das and Roy 1984).

31. Data for permeability of the anhydrite composites suggested that the interfacial region can act as a preferred pathway for fluid transfer, but does not as long as the integrity of the anhydrite is maintained (anhydrite

solubility was a problem in some tests). We achieved permeability values on the order of  $10^{-6}$  darcies or lower, and anticipate better bonding from curing under restraining (field) conditions.

32. Interfacial regions of smaller composite samples with all three rock types revealed limited diffusion of cations through the grout, anhydrite, or halite near the contact. Observations of sample surfaces after bond-strength tests, and of interfaces in thin section, revealed that gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) or  $\text{Ca}(\text{OH})_2$  crystallized along interfaces between anhydrite and cement. Although this could threaten seal integrity, most observations and test results indicate that acceptable compatibility between grout and host rock was achieved. SEM observations of anhydrite with the salt-containing mixture suggested that compatibility was not improved significantly through use of this salt-free formulation. However, all test results suggested the likelihood of attaining durable sealing of repository access shafts by the use of such chemically tailored cementitious mixtures.



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Table 1  
Components of Cementitious Mixtures Used  
for Interfacial Experiments

Component (Wt % of Total)	Mixture	
	83-05	83-06
Class H cement	28.93	30.66
Class C fly ash	9.72	19.39
SiO <sub>2</sub> flour	5.97	6.29
CaSO <sub>4</sub> additive	3.41	3.62
NaCl	5.17	--
Plasticizer	0.06	1.01
Defoamer	0.10	0.11
Deionized water	16.84	16.39
Sand	29.80	31.62

Table 2

## Physical and Mechanical Properties of Mixtures 83-05 and 83-06

Property	Mixture	
	83-05	83-06
Porosity at 28 days, %	22.8	15.8
Permeability, darcies		
7 days	$2.98 \times 10^{-8}$	$<10^{-8}$
14	$<10^{-8}$	$<10^{-8}$
28	$<10^{-8}$	$<10^{-8}$
Thermal conductivity, W/m·K		
14	--	$1.37 \pm 0.15$
28	--	$1.23 \pm 0.10$
Dynamic modulus, GPa		
7 days	--	$27.6 \pm 0.3$
14	$23.6 \pm 0.5$	$30.5 \pm 0.3$
28	--	$35.0 \pm 0.3$
Tensile strength, MPa		
28 days	--	$2.5 \pm 0.4$
56		$3.8 \pm 0.9$
28**		$2.9 \pm 0.6$
56†		$4.9 \pm 0.1$
Compressive strength, MPa		
7 days	$32.3 \pm 1.7$	$53.5 \pm 4.7$
14	$39.1 \pm 0.1$	$75.7 \pm 3.3$
28	$53.1 \pm 2.6$	$84.7 \pm 8.0$

\* Cured at high humidity.

\*\* Cured in E33 simulated groundwater.

† Direct tensile test.

Table 3  
Physical Properties of Rock Materials

<u>Rock Type</u>	<u>File No.</u>	<u>Porosity, %</u>	<u>Water Permeability, darcies</u>
Anhydrite	C76	0.0*	$<10^{-8}$
Siltstone	C79	5.0	$>10^{-4}$ **

\*  $<0.01$  percent.

\*\* Not precisely quantified due to rapid flow.

Table 4  
Bond Strength, Mixture 83-06 with Anhydrite,  
Cured at 38 C

<u>Curing Time, days*</u>	<u>Bond Strength, MPa (<math>\pm</math> Standard Deviation)</u>
14	$1.5 \pm 0.4$
28	$1.5 \pm 0.2$
28**	$0.6 \pm 0.2$
56	$0.8 \pm 0.5$
56**	$1.1 \pm 0.1$
90	$0.5 \pm 0.2$

\* Samples cured at  $>95$  percent relative humidity except where noted.

\*\* Cured in E33 simulated groundwater.

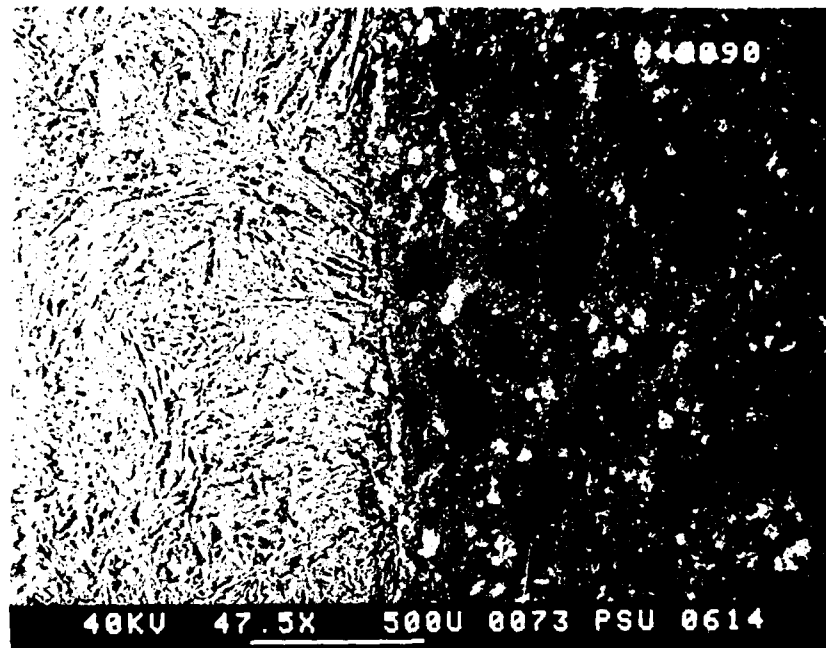


Figure 1. SEM back-scatter image of bond between 83-05 grout and halite from No. 1 J. Friemel core

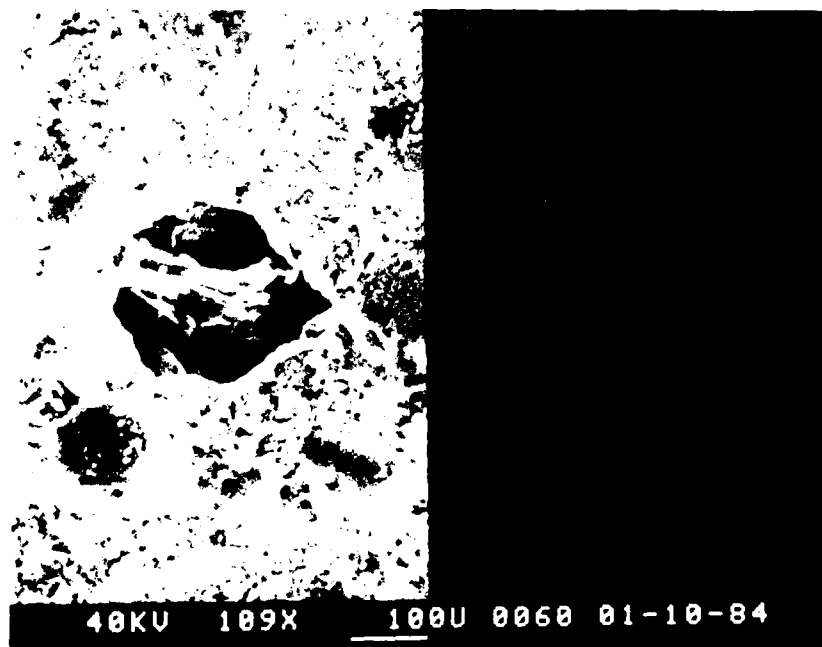


Figure 2. SEM image and corresponding elemental map of  $\text{Cl}^-$  distribution in 83-05 grout near interface with C75 halite

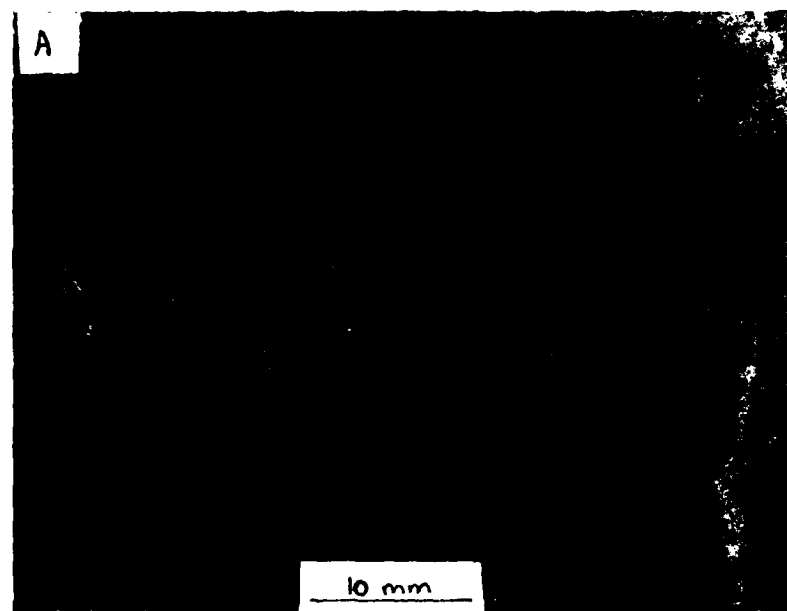


Figure 3. Separated interfacial surfaces of anhydrite (A) and 83-06 grout, showing flow path of permeability test

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